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JIS G 1328 : 2006

Methods for chemical analysis of ferroniobium

(Amendment 1)

JIS G 1328:1982 has been revised under date of February 20, 2006. The revised items are included in this Amendment 1.

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Foreword

This translation has been made based on the Amendment to the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law.
Consequently, **JIS G 1328**:1982 is partially replaced with this Amendment.

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Methods for chemical analysis of ferroniobium (Amendment 1)

JIS G 1328:1982 is revised as follows.

8.3.3 Apparatus, Devices and Materials

Replace (1) Oxygen Refining Part with the following.

(1) **Oxygen refining part** This part shall be in accordance with 8.2.3 (1), as appropriate. However, for absorption agent of sulfur oxide soda lime shall be used, and for dehydration agent, magnesium perchlorate be used.

8.6.2 Apparatus, Devices and Materials

Replace (1) Oxygen Refining Part with the following.

(1) **Oxygen refining part** This part shall be in accordance with 8.2.3 (1), as appropriate. However, in sulfur oxide absorption tube, soda lime shall be filled, and in dehydration tube magnesium perchlorate, be filled respectively. Oxygen shall be fed to circulation loop at a definite pressure by pressure regulator.

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JAPANESE INDUSTRIAL STANDARD J I S

Methods for Chemical Analysis of Ferroniobium G 1328-1982

1. Scope

This Japanese Industrial Standard specifies methods for determination of niobium, tantalum, carbon, silicon, phosphorus, sulfur, tin, and aluminum in ferroniobium.

2. General Matters

The general matters common to determination methods shall be in accordance with JIS G 1301.

3. Methods for Determination of Niobium

3.1 Division of Methods The methods for determination of niobium shall be in accordance with either one of the following:

(1) Hydrolysis Separation Niobium Pentaoxide Gravimetric Method This method applies to the whole range of ferroniobium.

(2) Ion-Exchange Separation Niobium Pentaoxide Gravimetric Method This method applies to the whole range of ferroniobium.

3.2 Hydrolysis Separation Niobium Pentaoxide Gravimetric Method

3.2.1 Summary Decompose the sample with hydrofluoric acid and nitric acid, and apply white fume treatment with sulfuric acid. Dissolve this in hydrochloric acid and hydrogen peroxide water, and hydrolyze niobium and tantalum (including a part of titanium) with sulfurous acid and tannic acid. Filter out the precipitate to incinerate, dissolve mixed oxides of niobium, tantalum, etc. with hydrofluoric acid and sulfuric acid, and precipitate niobium, tantalum, etc. again with sulfurous acid and tannic acid. Fuse the mixed oxides with potassium pyrosulfate, dissolve it in ammonium oxalate, and obtain the amount of titanium dioxide according to diantipyril methane absorptiometry or Alyzarin S absorptiometry. Separately, obtain the amount of tantalum from this solution according to 4.3 Pyrogallol Absorptiometry or 4.4 Malachite Green Absorptiometry. Subtract the mass of oxides of titanium and tantalum from the mass of previously obtained mixed oxides.

3.2.2 Reagents The reagents shall be as follows:

(1) Hydrochloric acid

(2) Hydrochloric acid (1+1, 1+50)

(3) Nitric acid (1+1)

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(4) Hydrofluoric acid

(5) Sulfuric acid (1+1)

(6) Sulfurous acid water (saturated, about 6 %)

(7) Hydrogen peroxide water

(8) Potassium pyrosulfate

(9) Stannous chloride solution Dissolve 100 g of stannous chloride ($SnCl_2 \cdot 2H_2O$) in 250 ml hydrochloric acid and dilute with water to 500 ml.

(10) Niobium·tantalum mixed solution Weigh out 0.186 g of niobium pentaoxide (purity 99.5 % min.) and 0.017 g of tantalum pentaoxide (purity 99.5 % min.), transfer into a platinum or quartz crucible (30 ml), mix 5 g of potassium pyrosulfate and fuse by heating. After cooling, transfer the crucible into a beaker (300 ml), add about 150 ml ammonium oxalate solution [3.2.2 (12)] to dissolve by heating, and take out the crucible with washing by ammonium oxalate solution [3.2.2 (12)]. After cooling to ordinary temperature, transfer this solution into a 250 ml measuring flask, and dilute with ammonium oxalate solution [3.2.2 (12)] up to the marked line. 25 ml of this solution contains niobium and tantalum equivalent to the amount of separately taken with treated 0.200 g of ferroniobium of 65 % in niobium content and 7 % in tantalum content. For the preparation of this solution, prepare niobium solution and tantalum solution shown in 4.3.3 (2) and (6) separately, and the required amounts may be mixed.

(11) Tannic acid ($C_{14}H_{10}O_9$)

(12) Ammonium oxalate solution Dissolve 40 g ammonium oxalate [$(NH_4)_2C_2O_4 \cdot H_2O$] in water to make 1000 ml.

(13) Diantipyril methane solution Dissolve 15 g diantipyril methane [$CH_2[C_3N_2O(CH_3)_2C_6H_5]_2 \cdot H_2O$] in 300 ml water and 30 ml sulfuric acid (1+1) by heating, cool to ordinary temperature, dilute with water to 1000 ml, and store in a brown bottle.

(14) Alizarin S solution Dissolve 0.2 g of sodium alizarin sulfonate ($C_{14}H_7O_7S \cdot Na \cdot H_2O$) in water and dilute with water to 100 ml.

(15) Standard titanium solution (20 μg TiO_2/ml) Weigh out 0.2000 g of titanium dioxide (purity 99.0 % min.) which is cooled after igniting at 900 to 950°C preliminarily for about 30 min, transfer into a platinum or quartz crucible, mix with 5 g potassium pyrosulfate to fuse by heating. After cooling, transfer the crucible into a beaker (500 ml), add about 300 ml ammonium oxalate solution [3.2.2 (12)], dissolve by heating, take out the crucible with washing by ammonium oxalate solution, cool to ordinary temperature, transfer into a 500 ml measuring flask, and dilute with ammonium oxalate solution up to the marked line to take it as the standard stock solution (400 μg TiO_2/ml). At each time of use, dilute a part of this standard solution with ammonium oxalate solution correctly to 20 times to take it as the standard titanium solution.

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3.2.3 Weighing-Out Amount of Sample Weigh out the sample by 0.20 g to the nearest 0.1 mg.

3.2.4 Operation The determination operation shall be carried out according to the following procedures:

(1) Decomposition of Sample Transfer the weighed-out sample [3.2.3] into a platinum dish (No. 100), cover it, add 10 ml hydrofluoric acid, after decomposing by droping 5 ml nitric acid (1+1), add 5 ml sulfuric acid (1+1), heat to generate sulfuric acid white fume for 7 to 8 min([1]) and cool it.

Note ([1]) If generate white fume violently, errors may be caused by sputtering and the like, and therefore cares shall be taken to white fume treatment.

(2) Separation of Mixed Oxides Transfer the contents in a platinum dish obtained in 3.2.4 (1) while washing with water into a beaker (500 ml) containing preliminarily 2 ml of hydrogen peroxide water, further add 1 ml hydrogen peroxide water in the platinum dish to dissolve adhering matters completely and wash with water to join with the previous beaker. Dilute this with warm water to about 250 ml, after adding 15 ml hydrochloric acid, add 80 ml sulfurous acid water (saturated, about 6 %) while mixing with stirring, when precipitates have begun to generate add about 1 g tannic acid and a small amount of filter paper to heat([2]), and after boiling for about 10 min, stand it still in running water for 1 h or more.

Filter out the precipitates by using filter paper (class 6)([3]), and wash with hydrochloric acid (1+50) 5 to 6 times. Dry the precipitates together with the filter paper, transfer to a porcelain crucible (30 ml), and incinerate by heating to dark red colour. Transfer this incineration residue into a platinum dish (No. 100)([4]), add 10 ml hydrofluoric acid and 5 ml sulfuric acid (1+1), heat to generate sulfuric acid white fume for 7 to 8 min([1]), and cool it.

Transfer the contents in this platinum dish into a beaker (500 ml) put with 2 ml hydrogen peroxide water preliminarily while washing with water, further add 1 ml hydrogen peroxide water into the platinum dish to dissolve completely the adhering substances, and wash with water to joint to the previous beaker. Dilute this with warm water to about 250 ml, after adding 15 ml hydrochloric acid, add while mixing with stirring 80 ml sulfurous acid water (saturated, about 6 %), when precipitates have begun to generate, add about 1 g of tannic acid and a small amount of filter paper pulp to heat, and boil for about 10 min. After cooling this in running water to ordinary temperature or under, filter the precipitates by using filter paper (class 6)([3]), and wash with hydrochloric acid (1+50) 5 times or more. Discard the filtrate and washings.

Notes ([2]) After generation of precipitate, filter paper pulp may be added. In this case, it is required to filter after mixing with stirring sufficiently.

([3]) In the case where the amount of precipitate is large, several funnels shall be used.

([4]) When oxides have remained in the crucible, dissolve with a small amount of hydrofluoric acid and transfer into a platinum dish.

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(3) Weighing of Mixed Oxides Transfer the precipitates obtained in 3.2.4 (2) together with the filter paper into a platinum crucible (No. 30), gradually increase the heating temperature to incinerate, thereafter ignite at 900 to 1000°C to make constant quantity, stand to cool to ordinary temperature in a desiccator and weigh the mass to take it as the quantity of mixed oxides (w_1).

(4) Preparation of Correction Solution Mix 5 g potassium pyrosulfate in mixed oxides obtained in 3.2.4 (3) and heat to fuse. After standing cool, transfer the platinum crucible into a beaker (200 ml), add about 50 ml ammonium oxalate solution [3.2.2 (12)], heat the melt to dissolve, take cut the platinum crucible after washing with ammonium oxalate solution, cool to ordinary temperature, transfer into a 100 ml measureing flask and dilute with ammonium oxalate solution up to the marked line.

(5) Determination of Titanium Dioxide for Correction

(a) Separately take 10 ml from the correction solution obtained in 3.2.4 (4), transfer into a 50 ml measuring flask, add 14 ml hydrochloric acid (1+1) and mix with shaking.

(b) Add 20 ml diantipyril methane solution [3.2.2 (13)] to this and dilute with water up to the marked line. After standing as it is for 20 min or more, transfer a part of it into an absorption cell of photometer and measure the absorbance at near 385 nm in wavelength.

(c) Insert this absorbance in the working curve([5]) and obtain the amount of titanium dioxide (w_2).

Note ([5]) Preparation of Warking Curve for Determination of Titanium Dioxide Prepare several 50 ml measuring flasks, stepwise add accurately 0 to 15 ml standard titanium solution (0 to 300 μg as titanium dioxide), make respective liquid amount 15 ml with ammonium oxalate solution [3.2.2 (12)], and add 14 ml hydrochloric acid (1+1) to mix with shaking. Hereafter, operate according to the procedure of 3.2.4 (5)(b) and measure the absorbance. Obtain the relation between the absorbance and the amount of titanium dioxide in coloured solution to take it as the working curve.

(6) Determination of Tantalum Pentaoxide Obtain the tantalum content (w_3) according to the tantalum determination method of 4.3 or 4.4.

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3.2.5 Calculation Calculate the niobium content in sample according to the following formula:

$$\text{Niobium (\%)} = \frac{[w_1-(W_2+W_3)]\times 0.6991}{W}\times 100$$

where w_1: mass of mixed oxides obtained in 3.2.4 (3) (g)

W_2: mass of titanium dioxide contained in the abovementioned (g)

$W_2 = 10\,w_2$

w_2: mass of titanium dioxide in the separately taken correction solution (g)

W_3: mass of tantalum pentaoxide contained in mixed oxides (g)

$W_3 = w_3 \times 0.01221 \times W$

w_3: tantalum content in sample (%)

W: amount of weighed out sample (g)

Remark: Determination of titanium dioxide for correction may be carried out according to the following procedures: Separately take 10 ml from the correction solution obtained in 3.2.4 (4) and transfer into a beaker (100 ml). Concentrate it by heating to make the amount of liquid about 5 ml, cool to ordinary temperature, transfer into a 50 ml measuring flask while washing with 20 ml hydrochloric acid and 10 ml ammonium oxalate solution [3.2.2 (12)], after cooling to 15°C or under. Add 3.5 ml Alyzarin S solution [3.2.2 (14)] and 10 ml stannous chloride solution [3.2.2 (9)] in sequence, and dilute with hydrochloric acid (1+1) up to the marked line(6). Maintain this solution at about 15°C, after standing still for 30 min, transfer a part of it into an absorption cell of photometer, and measure the absorbance at near 760 nm in wavelength using the blank test solution(7) as the contrast solution. Insert this absorbance in the working curve(8) to obtain the amount of titanium dioxide (w_2).

Notes (6) Make the hydrochloric acid concentration of final sample solution 6 N.

(7) The blank test solution is the solution treated similarly to the sample over the whole operation excluding only the sample.

(8) The working curve in this case shall be prepared as follows: Prepare several beakers (100 ml), add respectively 25 ml accurately niobium·tantalum mixed solution [3.2.2 (10)]. Stepwise add to these 0 to 10 ml standard titanium solution (0 to 200 μg as titanium dioxide). Concentrate these solutions by heating to 5 ml in liquid amount, cool to ordinary temperature, transfer into a 50 ml measuring flask while washing with 20 ml hydrochloric acid and 10 ml ammonium oxalate solution [3.2.2 (12)] and cool to 15°C or under. Sequentially add to thse correctly 3.5 ml Arizarin S solution [3.2.2 (14)] and 10 ml stannous chloride solution [3.2.2 (9)], and dilute with hydrochloric acid (1+1) up to the marked line(6).

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Maintain these solutions at about 15°C, after standing still for 30 min, transfer a part of respective solutions into an absorption cell of photometer, and measure the absorbance at near 760 nm in wavelength using the solution of adding amount of standard titanium solution of 0 as the contrast solution. Obtain the relations between these absorbancies and the amounts of titanium dioxide in the coloured solution to take as the working curve.

3.3 Ion-Exchange Separation Niobium Pentaoxide Gravimetric Method

3.3.1 Summary Decompose a sample with suitable acid to make the solution of hydrofluoric acid and nitric acid. Pass this solution through ion-exchange resin column to adsorb niobium, tantalum, etc. in resin, after eluting iron and the like with eluate (A), separately elute only niobium with eluate (B). Adjust the acidity of this eluate with aqueous ammonia and ammonium acetate, precipitate niobium with tannic acid to filter out, incinerate by ignition and weigh the mass.

3.3.2 Applicable Field In the cases where the sample is decomposed easily and where the decomposition is incomplete, the preparation of sample solution shall be carried out respectively with different operation.

3.3.3 Reagents The reagents shall be as follows:

(1) Nitric acid (1+1)

(2) Hydrofluoric acid

(3) Sulfuric acid (1+1)

(4) Aqueous ammonia (1+1)

(5) Eluate (A) Put 308 ml nitric acid and 296 ml hydrofluoric acid into a 1000 ml polyethylene measuring flask to dilute with water up to the marked line and transfer into a polyethylene bottle to store.

(6) Eluate (B) Put 308 ml nitric acid and 37 ml hydrofluoric acid into a 1000 ml polyethylene measuring flask to dilute with water up to the marked line and transfer into a polyethylene bottle to store.

(7) Ammonium nitrate solution Dissolve 20 g ammonium nitrate in about 900 ml water, adjust pH at 6.5 ± 0.5 with aqueous ammonia (1+1) and dilute with water to 1000 ml.

(8) Tannic acid solution Dissolve 10 g tannic acid ($C_{14}H_{10}O_9$) in 90 ml ethyl alcohol (30 v/v %) and adjust pH at 6.5 ± 0.5 with aqueous ammonia.

(9) Ammonium acetate (CH_3COONH_4) solution (10 w/v%)

(10) Methyl Red solution Dissolve 0.1 g of Methyl Red [$(CH_3)_2NC_6H_4N{:}NC_6H_4{\cdot}COOH$] in 50 ml ethyl alcohol (95 v/v %) and dilute with water to 100 ml.

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3.3.4 Device Ion-exchange resin column (see Attached Fig. 1) Into a polyethylene tube with slendered one end of about 250 mm in length and about 11 mm in inner diameter, pack loosely the polypropylene wool loosened with water in about 5 mm thickness, flow-into 10 ml of strongly basic anion-exchange resin (74 to 149 μm) swelled with water to settle, and thereafter fill polypropylene wool in upper part to about 5 mm in thickness. Adjust the packing of polypropylene wool so that the flowing-out velocity of water from this ion-exchange resin column becomes 1.0 ± 0.2 ml/min. Further, use the new ion-exchange resin column after passing about 60 ml eluate (A) [3.3.3 (5)].

3.3.5 Weighing-Out Amount of Sample Weigh out the sample by 0.20 g to the nearest 0.1 mg.

3.3.6 Operation Determination operation shall be carried out according to the following procedures:

(1) Preparation of Sample Solution

(a) Sample to Be Decomposed Easily by Hydrofluoric Acid and Nitric Acid Transfer the weighed-out sample [3.3.5] into a polyethylene beaker (100 ml) or a platinum dish (No. 100) to cover, and after adding 8 ml hydrofluoric acid, drop 2 ml nitric acid (1+1) to decompose.

(b) Sample to Be Decomposed Incompletely by Hydrofluoric Acid and Nitric Acid Transfer the weighed-out sample [3.3.5] into a teflon beaker (100 ml) or a platinum dish (No. 100) to cover with lid, add 8 ml hydrofluoric acid, 2 ml nitric acid (1+1), and 1 ml sulfuric acid (1+1) to decompose and evaporate by heating to dryness. After standing to cool, add 8 ml hydrofluoric acid and 2 ml nitric acid (1+1) and heat at a low temperature to dissolve([9]).

Note ([9]) If the heating is continued too long time, the acid concentration changes, and therefore take so care that it is dissolved in as short time as possible.

(2) Ion-Exchange Separation Pass the sample solution obtained in 3.3.6 (1) through the ion-exchange resin column [3.3.4] which is passed-through with 30 ml eluate (A) [3.3.3 (5)].

Then, wash the lid and beaker with 10 ml eluate (A) and pass through the same ion-exchange resin column. After repeating this operation twice, at last pass eluate (A) respectively 10 ml three times, and receive the eluate in a polyehtylene beaker (300 ml) to store([10]). Then, pass 50 ml eluate (B) [3.3.3 (6)] dividing in several times through the ion-exchange resin column and receive the eluate in a teflon beaker (300 ml)([11]).

Notes ([10]) This eluate can be used for determination of aluminium, tin, etc. When these determination is not carried out, discard.

([11]) When carrying out determination of tantalum, store the ion-exchange resin column at that time.

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(3) Precipitation Separation Add aqueous ammonia (1+1) gradually to eluate obtained in 3.3.6 (2), at near a point of neutralization add 2 to 3 drops of Methyl Red solution as indicator, and further drop aqueous ammonia (1+1) until the solution turns yellow. After adding 15 ml ammonium acetate solution (10 w/v %), dilute with hot water to about 200 ml, heat to about 80 °C, while mixing with sterring add 50 ml tannic acid solution [3.3.3(8)], after heating in a water bath at about 80°C for about 1 h cool in running water to ordinary temperature or under, filter-out the precipitates(3) by using filter paper (class 5 B), and wash sufficiently with ammonium nitrate solution [3.3.3 (7)]. Discard the washings.

(4) Weighing Transfer the precipitates obtained in 3.3.6 (3) together with the filter paper into a platinum crucible (No. 30) of known mass to incinerate, thereafter ignite at 1000°C to constant mass, and cool to ordinary temperature in a desiccator to weigh the mass.

3.3.7 Calculation Calculate the niobium content in sample according to the following formula:

$$\text{Niobium } (\%) = \frac{(w_1 - w_2) \times 0.6991}{W} \times 100$$

where w_1: mass of platinum crucible put with niobium pentaoxide (g)

w_2: mass of platinum curcible (g)

W: amount of weighed-out sample (g)

4. Determination Methods for Tantalum

4.1 Division of Methods The determination method for tantalum shall be in accordance with either one of the following:

(1) Tantalum Pentaoxide Gravimetric Method This method applies to sample of 1 % or more in tantalum content.

(2) Pyrogallol Absorptiometry This method applies to sample of under 10 % in tantalum content.

(3) Malachite Green Absorptiometry This method applies to sample of under 1 % in tantalum content.

4.2 Tantalum Pentaoxide Gravimetric Method

4.2.1 Summary Decompose sample with suitable acid to prepare the solution of hydrofluoric acid and nitric acid. Adsorb niobium, tantalum, etc. in the resin by passing this solution through ion-exchange resin column, and after elution separation of niobium and the like separately elute tantalum by eluate (C). Adjust the acidity of this eluate by aqueous ammonia and ammonium acetate, precipitate tantalum by tannic acid to filter out and ignite to incinerate to weigh the mass.

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4.2.2 Reagents The reagents shall be as follows:

(1) Aqueous ammonia (1+1)

(2) Eluate (C) Take 240 g ammonium nitrate, 14 g ammonium hydrogen fluoride and 18 g ammonium fluoride in a polyethylene beaker, dissolve in about 900 ml water, and dilute with water until near 1000 ml. By using Bromocresol Green test paper adjust pH at 3.5 with nitric acid (1+1) or aqueous ammonia and dilute with water to 1000 ml. Store this solution in a polyethylene bottle.

(3) Ammonium nitrate solution Dissolve 20 g ammonium nitrate in about 900 ml water, adjust pH at 6.5 ± 0.5 with aqueous ammonia (1+1) and dilute with water to 1000 ml.

(4) Tannic acid solution Dissolve 10 g tannic acid ($C_{14}H_{10}O_9$) in 90 ml ethyl alcohol (30 v/v %) and adjust pH at 6.5 ± 0.5 with aqueous ammonia.

(5) Ammonium acetate solution (10 w/v %)

(6) Methyl Red solution Disolve 0.1 g Methyl Red [$(CH_3)_2NC_6H_4COOH$] in 50 ml ethyl alcohol (95 v/v %) and dilute with water to 100 ml.

4.2.3 Device The device shall be in accordance with 3.3.4.

4.2.4 Weighing Amount of Sample Weigh out 0.20 g sample to the nearest 0.1 mg.

4.2.5 Operation The determination operation shall be carried out according to the following procedures:

(1) Preparation of Sample Solution Operate in accordance with procedures of 3.3.6 (1).

(2) Ion-Exchange Separation Operate in accordance with procedures of 3.3.6 (2), pass respective 10 ml eluate (C)[4.2.2 (2)] through ion-exchange resin column which has been passed with eluate (B) five times, and collect the eluate in a polyethylene beaker (300 ml).

(3) Precipitation Separation Add gradually aqueous ammonia (1+1) to the eluate obtained in 4.2.5 (2), add 2 to 3 drops of Methyl Red solution as indicator at near point of neutralization, and further drop aqueous ammonia (1+1) until the solution turns yellow. After adding 15 ml ammonium acetate solution (10 w/v %), dilute with hot water to about 200 ml, heat to about 80°C, add 50 ml tannic acid solution [4.2.2 (4)] while mixing with stirring, after heating about 1 h in a water bath at about 80°C cool to ordinary temperature or under in running water, filter the precipitates by using filter paper (class 5 B), and wash sufficiently with ammonium nitrate solution [4.2.2 (3)]. Discard the washings.

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(4) Weighing Transfer the precipitates obtained in 4.2.5 (3) together with the filter paper into a platinum crucible (No. 30) of known mass to incinerate, then ignite at 1000°C to constant mass, and cool to ordinary temperature in a desiccator to weigh the mass.

4.2.6 Calculation Tantalum content in sample shall be calculated according to the following formula:

$$\text{Tantalum } (\%) = \frac{(w_1 - w_2) \times 0.8190}{W} \times 100$$

where w_1: mass of platinum crucible put with tantalum pentaoxide (g)

w_2: mass of platinum crucible (g)

W: weighed-out amount of sample (g)

4.3 Pyrogallol Absorptiometry

4.3.1 Summary (In the case of hydrolytic separation) Decompose the sample with hydrofluoric acid and nitric acid, white-fume treate with sulfuric acid to dissolve in hydrochloric acid and hydrogen peroxide water, hydrolyze tantalum together with niobium and the like with sulfurous acid and tannic acid and filter out. Redissolve this precipitate and repeat the hydrolysis operation. Filter the precipitate to incinerate, fuse with potassium pyrosulfate and dissolve in ammonium oxalate. (In the case of ion exchange separation) Decompose the sample with suitable acid to make solution of hydrofluoric acid and nitric acid, adsorb tantalum together with niobium and the like, elute niobium and the like, thereafter elute tantalum with eluate (C). White-fume treat this elute with sulfuric acid to dissolve in ammonium oxalate.

Colourate tantalum as pyrogallol complex from the solution separated of interfering component according to either one of the methods, and measure the absorbance.

4.3.2 Applicable Field In the cases of hydrolysis separation and ion-exchange separation, the preparation of sample solution and preparation of working curve shall be carried out respectively according to different operation.

4.3.3 Reagents The reagents shall be as follows:

(1) Sulfuric acid (1+1)

(2) Niobium solution Weigh out 0.372 g niobium pentaoxide (99.5 % or more in purity), transfer into a platinum or quartz crucible (30 ml), mix 5 g potassium pyrosulfate and heat to fuse. After standing to cool, transfer the crucible into a beaker (500 ml), add 300 ml ammonium oxalate solution [4.3.3 (3)], and heat to dissolve the melt. Take out the crucible with washing with ammonium oxalate solution, cool to ordinary temperature, transfer into a 500 ml measuring flask, and dilute with ammonium oxalate solution up to the marked line. 25 ml of this solution contains 13 mg niobium, and it corresponds to amount of niobium in the solution separately taken by its 1/10 with treating 0.2 g ferroniobium of niobium content 65 %.

(3) Ammonium oxalate solution The solution shall be in accordance with 3.2.2 (12).

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(4) Pyrogallol solution Dissolve 30 g pyrogallol ($C_6H_6O_3$) in water containing 6.7 ml phosphoric acid and dilute with water to 100 ml. Prepare this solution at each use.

(5) Standard titanium solution The solution shall be in accordance with 3.2.2 (15).

(6) Standard tantalum solution (100 μg Ta/ml) Weigh out 0.1527 g of tantalum pentaoxide (99.5 % or more in purity) transfer into a platinum crucible (No. 30), mix 5 g potassium pyrosulfate and heat to fuse. After standing cool transfer the crucible into a beaker (300 ml), add 150 ml ammonium oxalate solution [4.3.3 (3)], heat to dissolve the melt and take out the platinum crucible with washing with ammonium oxalate solution. Cool to ordinary temperature, transfer the solution into a 250 ml meassuring flask, and dilute with ammonium oxalate solution up to the marked line to take it as the standard stock solution. At each time of use, dilute a part of this standard solution with ammonium oxalate solution correctly to 5 times to take it as the standard tantalum solution.

4.3.4 Weighing-Out Amount of Sample Weigh out 0.20 g sample to the nearest 0.1 mg.

4.3.5 Operation The determination operation shall be carried out according to the following procedures:

(1) Preparation of Sample Solution

(a) In the Case of Hydrolysis Separation Use the solution obtained in 3.2.4 (4).

(b) In the Case of Ion-Exchange Separation Transfer the solution obtained in 4.2.5 (2) into a platinum dish (No. 100), add 1 ml sulfuric acid (1+1), heat to vaporize ammonium salts, and when the white fume of sulfuric acid has begun to generate, leave to cool. Add 2 ml sulfuric acid (1+1), and after washing the inner wall of platinum dish with water, heat to evaporate until white fume of sulfuric acid begins to generate. After standing to cool, add 30 ml ammonium oxalate solution [4.3.3 (3)], heat at a low temperature to dissolve, cool to ordinary temperature, transfer into a 100 ml measuring flask, and dilute with ammonium oxalate solution up to the marked line.

(2) Colouration

(a) Separately take 10 ml sample solution obtained in 4.3.5 (1), transfer into a 100 ml measuring flask, and add 40 ml ammonium oxalate solution [4.3.3 (3)] to make it at a definite liquid temperature([12]).

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(b) Add 30 ml pyrogallol solution [4.3.3 (4)] at the same temperature as that of this solution, mix with shaking, keep the liquid temperature same as the previous liquid temperature for about 15 min, and dilute with ammonmoxalate solution at the same temperture up to the marked line.

Note ([12]) Because the absorbance of pyrogallol complex of tantalum is influenced by liquid temperature too much, it shall be made the same condition as the temperature at the time of preparation of working curve.

(3) Measurement of Absorbance Immediately take a part of solution obtained in 4.3.5 (2) into an absorption cell of photometer, and measure the absorbance ([13]) at near 430 nm in wavelength using blank test solution as the contrast solution.

Notes ([13]) In the case where the solution of 4.3.5 (1)(a) (in the case of hydrolysis separation) is colourated, insert amount of titanium dioxide (w_2) in the mixed oxides obtained in 3.2.4(5) or Remark in the working curve for correction([14]) and it is required to subtract the corresponding absorbance.

([14]) Working curve for titanium correction shall be prepared as follows:

Prepare several 100 ml measuring flasks, respectively add 25 ml niobium solution [4.3.3 (2)] accurately, add to these stepwise 0 to 15 ml standard titanium solution (0 to 300 μg as titanium dioxide), dilute with ammonium oxalate solution [4.3.3 (3)] to 50 ml and make it at a difinite temperature. Hereafter operate according to the procedures on and after 4.3.5 (2) (b), and measure the absorbance. Obtain the relation between the absorbance and the amount of titanium dioxide in the colourated solution and take it as the working curve.

4.3.6 Preparation of Working Curve The working curve shall be prepared as follows:

(1) Working Curve in the Case of Hydrolysis Separation Prepare several 100 ml measuring flasks, add respectivly 25 ml niobium solution [4.3.3 (2)] accurately, stepwise add to these 0 to 20 ml standard tantalum solution accurately (0 to 2.00 mg as tantalum), and dilute with ammonium oxalate solution [4.3.3 (3)] to 50 ml. Hereafter, operate according to the procedures on and after 4.3.5 (2)(b), and measure the absorbance. Obtain the relation between the absorbance and the amount of tantalum in colourated solution and take it as the working curve.

(2) Working Curve in the Case of Ion-Exchange Separation Prepare several 100 ml measuring flasks, add stepwise 0 to 20 ml standard tatalum solution (0 to 2.00 mg as tantalum) accurately, and dilute with ammonium oxalate solution [4.3.3 (3)] to 50 ml to make at a definite temperature. Hereafter, operate according to the procedures on and after 4.3.5 (2)(b) to measure the absorbance. Obtain the relation between the absorbance and the amount of tantalum in the colourated solution and take it as the working curve.

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4.3.7 Calculation Insert the absorbance obtained in 4.3.5 (3), when the solution of 4.3.5(1) (a) (in the case of hydrolysis separation) is colourated in the working curve prepared in 4.3.6 (1), and when the solution of 4.3.5 (1)(b) (in the case of ion-exchange separation) is colourated in the working curve prepared in 4.3.6 (2) to obtain the amount of tantalum and calculate the tantalum content in the sample according to the following formula:

$$\text{Tantalum } (\%) = \frac{A}{W \times B} \times 100$$

where A : amount of tantalum in the separately taken sample solution (g)

W : weighed-out amount of sample (g)

B : aliquot ratio of sample solution. It is 1/10 herein.

4.4 Malachite Green Absorptiometry

4.4.1 Summary (In the case of hydrolysis separation) Decompose sample with hydrofluoric acid and nitric acid, white-fume treat with sulfuric acid to dissolve in hydrochloric acid and hydrogen peroxide water, hydrolyze tantalum together with niobium and the like with sulfurous acid and tannic acid and filter. Redissolve this precipitate and repeat hydrolysis operation. Filter the precipitate to incinerate, fuse with potassium pyrosulfate and dissolve in ammonium oxalate solution.

(In the case of ion-exchange separation) Decompose sample with a suitable acid to make solution of hydrofluoric acid and nitric acid, adsorb tantalum together with niobium and the like in the ion-exchange resin, and after eluting niobium and the like elute tantalum with eluate (C). White-fume treat this eluate with sulfuric acid and dissolve in ammonium oxalate solution.

From the solution in which the interfering component are separated by either method make the tantalum as Malachite Green complex and measure its absorbance.

4.4.2 Field of Application In the case of hydrolysis separation and in the case of ion-exchange separation, the preparation method of sample solution shall be in accordance with other operation respectively.

4.4.3 Reagents The reagents shall be as follows:

(1) Hydrofluoric acid (1+17)

(2) Sulfuric acid (1+9)

(3) Ammonium oxalate solution The solution shall be in accordance with 3.2.2 (12).

(4) Malachite Green ($C_{23}H_{25}N_2Cl$) solution (0.2 w/v %)

(5) Benzene (C_6H_6)

(6) Standard tantalum solution (0.100 mg Ta/ml) The solution shall be in accordance with 4.3.3 (6).

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4.4.4 Weighing-Out Amount of Sample Weigh out 0.20 g sample to the nearest 0.1 mg.

4.4.5 Operation Determination operation shall be carried out according to the following procedures:

(1) Preparation of Sample Solution

(a) In the Case of Hydrolysis Separation Use the solution obtained in 3.2.4 (4).

(b) In the Case of Ion-Exchange Separation Use the solution obtained in 4.3.5 (1)(b).

(2) Colouration From the sample solution obtained in 4.4.5 (1) separately take depending upon the tantalum content 5 ml in the case of less than 0.5 %, and 2 ml in the case of not less than 0.5 %, transfer into a polyethylene separatory funnel (100 ml), add accurately 2 ml sulfuric acid (1+9) and 4 ml hydrofluoric acid (1+17) to mix with shaking, then add accurately 1.5 ml Malachite Green solution (0.2 w/v %) to mix with shaking, immediately add 20 ml benzene accurately and after mixing with violently shaking for 20 sec stand still to discard the lower layer of water solution.

(3) Measurement of Absorbance Filter the organic phase obtained in 4.4.5 (2) with a dry filter paper (class 5 A), discard the first filtrate, take a part of filtrate from the next filtrate into an absorption cell of photometer, and measure the absorbance at near 635 nm in wavelength by using benzene as the contrast solution.

4.4.6 Preparation of Working Curve The working curve shall be prepared as follows:

Prepare several 100 ml measuring flasks, add stepwise 0 to 20 ml standard tantalum solution (0 to 2.00 mg as tantalum) accurately, and dilute with ammonium oxalute solution [4.4.3 (3)] up to the marked line. Separately take from this solution 5 ml, transfer into a polyethylene separatory funnel (100 ml), add accurately 2 ml sulfuric acid (1+9) and 4 ml hydrofluoric acid (1+17) to mix with shaking, then add accurately 1.5 ml Malachite Green solution (0.2 w/v %) to mix with shaking, immediately add accurately 20 ml benzene and after mixing with violently shaking for 30 sec, stand still to discard the lower layer of water solution. Hereafter, measure the absorbance according to the procedure of 4.4.5 (3). Obtain the relation between the absorbance and the amount of tantalum in the coloured solution and take it as the working curve.

4.4.7 Calculation Insert the absorbance obtained in 4.4.5 (3) in the working curve prepared in 4.4.6 to obtain the amount of tantalum, and calculate the tantalum content in the sample according to the following formula:

$$\text{Tantalum (\%)} = \frac{A}{W \times B} \times 100$$

where A: amount of tantalum in the separately taken sample solution (g)

W: amount of weighed-out sample (g)

B: aliquot ratio of sample solution

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5. Carbon Determination Methods

5.1 Division of Methods Determination method of carbon shall be in accordance with either one of the following:

(1) Gas Volumetric Method This method applies to sample of not less than 0.05 % in carbon content.

(2) Conductivity Method This method applies to sample of not less than 0.001 % in carbon content.

(3) Coulometric Method This method applies to sample of not less than 0.001 % in carbon content.

(4) Infrared Absorption Method This method applies to sample of not less than 0.001 % in carbon content.

5.2 Gas Volumetric Method

5.2.1 Summary Heat the sample in a oxygen stream, oxidize sufficiently carbon to make carbon dioxide, collect this together with oxygen in a burete to measure the gas volume, then remove carbon dioxide by absorbing in alkali solution, and measure the remaining gas volume to obtain the volumetric reduction.

5.2.2 Apparatus and Device The apparatus and devices shall, as a rule, be in accordance with 5. and 6.3.2 of JIS Z 2615.

5.2.3 Weighing-Out Amount of Sample and Adding Amount of Ignition Improver Weigh out the sample depending upon carbon content in accordance with Table 1, as a rule, and add ignition improver. Select the most suitable ignition improver from those indicated in 5. (13) of JIS Z 2615, add the amount 2 to 4 times the sample to mix thoroughly or to cover on the sample.

Table 1. Weighing-Out Amount of Sample

Carbon content %	Weighing-out amount of sample
0.05 up to and excluding 10	2.0 g to the nearest 1 mg
0.10 up to and including 0.20	1.0 g to the nearest 1 mg

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5.2.4 Preliminary Operation The preliminary operation shall be in accordance with 6.3.3 of JIS Z 2615.

Further, in the case where tubular electric resistance heating furnace is used, the temperature in the combustion tube shall be kept at 1300 to 1400°C([15]).

Furthermore, in the case where a high-frequency induction heating furnace is used, conditions concerning high-frequency induction heating shall be set([16]).

Notes ([15]) Take care on the difference between the temperature indication of pyrometer and the temperature in the combustion tube to correct.

([16]) For example, it means conditions decided according to specifications of apparatus using anode current and grid current of high-frequency oscillator.

5.2.5 Determination Operation The determination operation shall be carried out according to the following procedures:

(1) In the Case of Using Tubular Electric Resistance Heating Furnace
The procedures shall be in accordance with 6.3.4 of JIS Z 2615.

(2) In the Case of Using High-Frequency Induction Heating Furnace
The procedures shall be in accordance with Remark in 6.3 of JIS Z 2615.

5.2.6 Blank Test The blank test shall be carried out according to the following procedures:

(1) In the Case of Using Tubular Electric Resistance Heating Furnace
The procedures shall be in accordance with 6.3.5 of JIS Z 2615.

(2) In the Case of Using High-Frequency Induction Heating Furnace
By using crucible put with the same amount of ignition improver as the amount added to the sample, excluding the sample the operation of 5.2.5 (2) shall be carried out.

Further, in the case where ignition improver which does not induce high-frequency is used, the operation is carried out with adding 0.5 to 1.0 g of iron or the like of known carbon content (as low as possible), and the carbon content in the added iron or the like are subtracted to take it as the blank test value.

5.2.7 Calculation The calculation shall be in accordance with 6.2.6 of JIS Z 2615.

5.3 Conductivity Method

5.3.1 Summary Heat the sample in oxygen stream, oxidize sufficiently carbon to make carbon dioxide, absorb in a definite amount of alkali solution, and measure the change of conductivity of alkali solution before and after absorption.

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5.3.2 Reagents The reagents shall be in accordance with 6.6.2 of JIS Z 2615.

5.3.3 Apparatus and Devices The apparatus and devices shall, as a rule, be in accordance with 5. and 6.6.3 of JIS Z 2615.

5.3.4 Weighing-Out Amount of Sample and Adding Amount of Ignition Improver Weigh out the sample of most suitable amount to the appratus to be used (usually 0.5 to 1.0 g) and add ignition improver. Select the most suitable ignition improver from those indicated in 5. (13) of JIS Z 2615, add the amount of 2 to 4 times that of sample, mix sufficiently or cover on the sample.

5.3.5 Preliminary Operation The preliminary operation shall be in accordance with 6.6.4 of JIS Z 2615.

Further, in the case where tubular electric resistance heating furnace is used, keep the temperature inside the combustion tube at 1300 to 1400°C([15]). Furthermore, in the case where high-frequency induction heating furnace is sued, set the conditions concerning high-frequency induction heating ([16]).

5.3.6 Determination Operation The determination operation shall be carried out according to the following procedures.

(1) In the Case of Using Tubular Electric Resistance Heating Furnace
The procedure shall be in accordance with 6.5.5 of JIS Z 2615.

(2) In the Case of Using High-Frequency Induction Heating Furnace

(a) Place the crucible put with sample and ignition improver on a receiving stand, and close the ignition improver tube. Feed oxygen at a specified flow rate to displace air in the tube, thereafter actuate the high-frequency induction heating furnace (the sample burns, carbon dioxide in combustion gas is absorbed in absorption liquid and the indicating value increases gradually.)

(b) When the recorder or indicator has indicated a definite value, read out the indicated value, break the high-frequency switch and take out the crucible.

5.3.7 Blank Test The blank test shall be carried out according to the following procedures:

(1) In the Case of Using Tublar Electric Resistance Heating Furnace
The procedures shall be in accordance with 6.6.6 of JIS Z 2615.

(2) In the Case of Using High-Frequency Induction Heating Furnace
By using a crucible put with ignition improver of the same amount as the amount added to the sample, excluding sample, carry out the operation of 5.3.6 (2).

Further, in the case where ignition improver which does not induce high-frequency is used, carry out the operation by adding 0.5 to 1.0 g of iron or the like of known carbon content (as low as possible), and subtract the carbon content in the added iron and the like to take it as the blank test value.

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5.3.8 Calculation The calculation shall be in accordance with 6.6.7 of JIS Z 2615.

5.4 Coulometric Method

5.4.1 Summary Heat the sample in oxygen stream, oxidize carbon sufficiently to make carbon dioxide, absorb it in weak alkaline balium salt solution at a definite pH, and measure the quantity of electricity required to return the decreased pH due to absorption to original pH by electrolysis of balium salt solution.

5.4.2 Reagents The reagents shall be in accordance with 6.7.2 of JIS Z 2615.

5.4.3 Apparatus and Devices The apparatus and devices shall, as a rule, be in accordance with 5. and 6.7.3 of JIS Z 2615.

5.4.4 Weighing-Out Amount of Sample and Adding Amount of Ignition Improver Weigh out the most suitable amount (usually 0.5 to 1.0 g) of sample relative to the apparatus to be used and add ignition improver. Select the most suitable ignition improver from those indicated in 5. (13) of JIS Z 2615, add the amount of ignition improver 2 to 4 times the amount of sample and mix thoroughly or cover on the sample.

5.4.5 Preliminary Operation The preliminary operation shall be in accordance with 6.7.4 of JIS Z 2615.

Further, in the case where tubular electric resistance heating furnace is used, keep the temperature inside the combustion tube at 1300 to 1400°C ([15]). Furthermore, in the case where high-frequency induction heating furnace is used, set the conditions concerning high-frequency induction heating([16]).

5.4.6 Determination Operation The determination operation shall be carried out according to the following procedures:

(1) In the Case of Using Tubular Electric Resistance Heating Furnace
The procedure shall be in accordance with 6.7.5 of JIS Z 2615.

(2) In the Case of Using High-Frquency Induction Heating Furnace

(a) Place the crucible put with sample and ignition improver on a receiving stand, and close the ignition improver tube. Feed oxygen at a specified flow rate to displace air in the tube, thereafter actuate the high-frequency induction heating furnace and at the same time return the indicating value to zero (when the sample burns, and absorption of carbon dioxide begins, the indication value increases gradually.)

(b) When the indicator has indicated a definite value, read out the indicated value, break the high-frequency switch and take out the crucible.

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